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(57) Abstract

This invention relates to a novel dry water-soluble and water-dispersible phytoactive formulation of glyphosate (the free acid N-phosphonomethylglycine) and an inorganic or organic, non-caustic, non-effervescing basic material.

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DRY WATER-DISPERSIBLE GLYPHOSATE PHYTOACTIVE FORMULATION

FIELD OF INVENTION

This invention relates to a novel dry water-dispersible, phytoactive formulation of glyphosate (the free acid N-phosphonomethylglycine) and an inorganic or organic, non-caustic, non-effervescing basic material.

DESCRIPTION OF THE PRIOR ART

Glyphosate, the free acid N-phosphonomethylglycine [$HOOCCH_2NH\ CH_2\ PO(OH)_2$] is well known in the art as an effective plant growth regulator, as an herbicide, as a defoliant or the like. Glyphosate is relatively water insoluble and is typically formulated as a water-soluble salt. These water-soluble salts are often difficult to obtain in solid form. They can form glassy, non-crystalline solids which transform rapidly into wet cakes when exposed to the air.

Commercial formulations of the water-soluble glyphosate salts are generally not sold in a solid form, but sold as aqueous solutions. Publications dealing with such formulations are:

U.S. Patent No. 3,799,758, Franz, March 26, 1974, entitled "N-Phosphonomethylglycine Phytotoxicant Compositions";

U.S. Patent No. 4,405,531, Franz, September 20, 1983, entitled "Salts of N-Phosphonomethylglycine";

U.S. Patent No. 4,315,765, Large, February 16, 1982, entitled "Trialkylsulfonium Salts of N-Phosphonomethylglycine and Their Use As Plant Growth Regulators and Herbicides";

U.S. Patent No. 4,397,676, Bakel, August 9, 1983, entitled "N-Phosphonomethyl Glycine Derivatives";

U.S. Patent No. 4,481,026, Prisbylla, November 6, 1984, entitled "Aluminum N-Phosphonomethylglycine And Its Use As A Herbicide";

Great Britain Publication Specification No. 2,098,481, entitled "Herbicidal Formulations".

In general, the aqueous glyphosate solutions contain from about 0.1 to about 70% free acid; more commonly they contain 25-35% glyphosate. These solutions are acidic and contain chelating properties.

To avoid the problems associated with aqueous solutions of glyphosate salt, it would be desirable to package and sell glyphosate compounds in dry or solid form. These formulations would realize substantial savings in terms of storage, transportation and container disposal charges and avoid the problem associated with aqueous solutions.

A number of patents generally disclosing so-called wettable or soluble powder compositions containing glyphosate salts include U.S. Patent No. 4,025,331; 4,414,158; 4,481,026 and 4,405,531. They broadly disclose compositions which include an inert solid extender and one or more surfactants. A disadvantage of such wettable powders is that the solid extender reduces the amount of active ingredient which can be

transported in a container of a particular size. A further disadvantage is that many of the phytoactive glyphosate-salt compounds are hygroscopic. They absorb water from the air and further they are deliquescent, wherein the compounds absorb atmospheric water and dissolve in the water thus absorbed.

A number of recent publications have described a solution to the problem of hygroscopicity or deliquescence.

U.S. Patent No. 4,140,513, Prill, February 20, 1979, entitled "Sodium Sesquiglyphosate" describes a unique salt of glyphosate which although water-soluble, crystallizes readily from concentrated aqueous solutions.

U.S. Patent No. 4,931, 080, Chan et al., June 5, 1990 entitled "Solid Phytoactive Compositions Method of Use and Methods Of Preparation" discloses the admixture of a phytoactive N-phosphonomethyl N-carboxymethyl compound, preferably a salt, with a solvent and a molten surfactant. The solvent is subsequently removed and the surfactant is cooled to a point wherein it becomes solid at ambient temperature. The product is further processed into particulate form.

U.S. Patent No. 5,047,079, Djafar et al., September 10, 1991, entitled "Method of Preparation and Use of Solid Phytoactive Compositions" describes phytoactive glyphosate composition as claimed in U.S. Patent No. 4,931,080, however, a more efficient process is claimed wherein one of the steps described in U.S. Patent No. 4,931,080 is eliminated, i.e., the elimination of the preparation of the salt in an aqueous solvent system.

EPA 378 985, entitled "Improved Glyphosate Formulations" discloses dry water soluble compositions comprising a water soluble salt of N-phosphonomethylglycine as a water-dispersible granule, water-soluble granule or water-soluble powder and additionally one or more liquid surfactants.

U.S. Patent No. 5,118,338, Moller, June 2, 1992, entitled "Herbicidal Formulation Containing Glyphosate Acid" discloses granular or powdery herbicidal formulations of the free acid, glyphosate and a surface-active ingredient that is a powdery or granular nonionic polyglycol ether of a straight chain, saturated, high molecular weight fatty alcohol.

SUMMARY OF THE INVENTION

A phytoactive, dry, water-soluble and water-dispersible formulation has now been developed which includes glyphosate, the free acid of N-phosphonomethylglycine and an inorganic or organic, non-caustic, non-effervescing basic material. The final product is a substantially non-hygro scopic formulation which retains its physical and chemical properties. While not necessary, other conventional adjuvants may be incorporated into the formulations.

It is an object of this invention to provide a dry, substantially non-hygroscopic, water-soluble and water-dispersible agriculturally acceptable formulation of glyphosate hereinafter described in more detail.

It is another object of this invention to provide a high-loading formulation of glyphosate which readily dissolves in water.

It is another object of the invention to provide a formulation with decreased dermal penetration thereby increasing user safety.

It is another object of this invention to provide a dry phytoactive formulation which can be packaged in low cost containers and is free from the problems associated with glyphosate salt solutions.

Another object of this invention is to provide a herbicidal method of use by applying said formulation to the locus.

A more thorough disclosure of the present invention is presented in the detailed description which follows:

DETAILED DESCRIPTION OF THE INVENTION

Accordingly, it has now been found that dry phytoactive glyphosate formulations can be readily prepared in a solid form that is readily solubilized in an aqueous media comprising the free acid, glyphosate, an inorganic or organic basic material and optionally surfactants or other adjuvants.

As used herein the term "solid" refers to the physical state wherein the formulation has a specific shape and volume and resists deformation. The solid may take the form of pellets, flakes, granules, powder or the like. The solid formulation may subsequently be dissolved in a suitable diluent, usually and preferably water, at a remote field site, and applied to the plants upon which the formulations phytoactivity is to be directed. The term "water-dispersible" is used in the broad sense to encompass water-soluble.

As used herein the term "phytoactive" as used in describing this invention means effective as a plant growth regulator, as a herbicide, as a defoliant or the like.

The term "herbicidally effective amount" designates any amount of the glyphosate disclosed herein which will kill a plant or any portion thereof. By "plants" is meant germinate seeds, emerging seedlings, and established vegetation including; roots and above-ground portions. Herbicidal effects include killing, defoliation, desiccation, stunting, leaf burn and dwarfing. Herbicidal effects are generally

achieved at higher application rates than growth regulating effects.

Several processes for the preparation of glyphosate are disclosed in the patent literature, e.g. U.S. Patent No. 3,799,758 (Franz, March 26, 1974).

The choice of a basic material is very important. The basic material must be a solid or liquid at ambient temperatures, preferably solid, non-hygroscopic and preferably non-effervescing.

Furthermore, it must give a basic pH in water. The preferred material to be used in the formulation of this invention is diammonium phosphate and basic guanidine salts for example, guanidinium acetate. Other basic materials which can be used in the practice of the invention include ammonium, sodium and potassium salts; urea derivatives such as
urea nitrate, urea phosphate; naturally occurring basic amino acids such as lysine, glycine, arginine and histidine; sugar alcohols such as sorbitol, guano and bone meal.

Suitable surfactants for use in this invention include nonionic, anionic, cationic, amphoteric surfactants and mixtures thereof.

While not intended to limit the invention, the following suitable surfactants are provided as examples. Nonionic surfactants include lauryl alcohols, for example, Brij 30; alkoxylated fatty alcohols, for example, PLURAFAC LF 120; block copolymers of EO/PO, for example, PLURONIC PE 10100 and SYNPERONIC PE/P84; alkylphenoxy polyethoxy ethanols, for example, TRITON X-100; sorbitan monopalmitates, for example, TWEEN 40; sorbitan trioleates, for example, TWEEN 85; polyalkoxylated alkyphenols, for example, WITCONOL NS 108 LQ; dialkylphenol ethoxylates, for example, IGEPAL DM

970 F; alkylpolyglysocides, for example, TRITON BG-10 AND APG 300 and mixtures thereof.

Cationic surfactants include primary fatty amines, for example, ARMEEN T; quaternary ammonium compounds, for example, ARQUADS-50; ethoxylated fatty diamines, for example, ETHODUOMEEN T/25 and ETHOMEEN T/25 and mixtures thereof.

Amphoteric surfactants include amino oxide and amino acid derivatives, for example, ARMEEN Z and betaine derivatives.

Other surfactants which can be used in the invention are generally classified as alkanolamides; ethoxylated alcohols; ethoxylated alkylphenols; ethoxylated amines and amides; ethoxylated fatty acids; fatty esters; glycol esters; quaternary surfactants; silicone based surfactants; sorbitan derivatives; sucrose and glucose esters and derivatives and sulfosuccinates and derivatives and mixtures thereof. These surfactants are listed in McCutcheon's Emulsifiers and Detergents which is incorporated herein by reference.

While the formulations need not include adjuvants, they can optionally contain conventional adjuvants such as wetting agents, dispersing agents, surfactants, anti-foaming agents, emulsifiers, drying acids, heat stabilizers, dispersants and other agriculturally acceptable materials. The choice of a particular adjuvant will be easily made by one skilled in the art, without undue experimentation based on the teachings of this specification. The term adjuvant is used herein to mean an agent used to aid the operation or improve the effectiveness of the phytoactivity of glyphosate.

Said formulations of this invention are typically in the range 1 to 99% weight glyphosate and preferably 20 to 99% weight glyphosate; 20 to 100% weight base preferably 35-100% weight base and 0.1 to 15% weight surfactant. The moisture content of said formulation is in the range from

about 0.01% to about 10% weight and preferably less than about 1.0% and most preferably less than about 0.3% weight.

The ratio of glyphosate to basic material varies over a wide range. The ratio by weight is typically from 10:1 to about 1:10. The most preferred ratio is from 4:1 to about 1:4. The most preferred ratio is from 1:1 to about 1:4.

The solid formulation of the invention may comprise a number of embodiments. Particularly, the formulation may comprise a wettable powder or water-soluble or water-dispersible granule.

Wettable powders are in the form of finely divided particles which disperse readily in water or other liquid carriers. The glyphosate particles are mechanically mixed together with the particles of basic material such as diammonium phosphate and surfactant. The mixture may be further milled to a fine powder in the range of about 50 mesh. Representative examples of the formulation are as follows. The formulations are based on percent by weight.

EXAMPLE 1

Glyphosate (90-100%)	20%
Diammonium phosphate	80%
Total	100%

EXAMPLE 2

Glyphosate (90-100%)	50%
Diammonium phosphate	50%
Total	100%

EXAMPLE 3

Glyphosate (90-100%)	45%
Diammonium phosphate	50%
IGEPAL DM 970	5%
Total	100%

EXAMPLE 4

Glyphosate (90-100%)	50%
Guanidinium acetate	45%
PLURONIC 25R8	5%
Total	100%

In the above examples, the glyphosate was milled with base, and IGEPAL DM 970 or PLURONIC 25R8 was optionally added to the mixture. After mixing, the mixture looked slightly soft. The mixture was kept overnight in an oven at 50°C. The final product was a dry brittle solid and was neither hygropscopic nor deliquescent.

The above formulation of this invention can be prepared in any suitable manner known to one skilled in the art.

In another embodiment, the glyphosate can be mixed with the surfactant and milled without the basic material. The range of surfactant is from about 0.1% to about 5.0% of the total dry weight. The basic material for example, diammonium phosphate or guanidinium acetate and any adjuvant may be added separately. This twin-pack is then available to the user to be tank-mix at the site of use.

Representative examples of this embodiment are as follows:

EXAMPLE 5

Component A	Glyphosate (90-100%)	95%
	PLURONIC F 88	5%
	Total	100%
Component B	Diammonium phosphate	100%

EXAMPLE 6

Component A	Glyphosate (90-100%)	95%
	PLURONIC F 68	5%
	Total	100%
Component B	Guanidinium acetate	100%

Another embodiment of the invention is a water-soluble or water-dispersible granular formulation. Granular formulations in general include both extrudates and relatively coarse particles and are usually applied without dilution to the area in which suppression of vegetation is desired. Materials used in the process of granulation may be in finely divided form for example an air-milled form. Granulation may be accomplished by those means well known in the art, including pan extrusion or agglomeration, fluidized beds, spray drying, drum dry and the like. A preferred method is the process disclosed in International Application Number PCT/AU 88/00201. The water-dispersible granules are formed by mixing the desired ingredients of the granules into an extrudable form, extruding the mix and then rolling the extrusion and optionally drying if required. This patent application is hereby incorporated by reference.

More particularly, the process comprises mixing the desired ingredients of the granule in the presence of water to form an extrudable wet mix, extruding the wet mix and rolling the wet extrusions to break down said extrusion to form the granules; and optionally drying the granules. The mixing step is carried out to form an extrudable wet mix which has a dough like consistency. After putting the mix in a form suitable for extrusion, extrusion takes place through orifices of specific sizes. The size of the granule is a function of the orifice size. Preferably extrusion orfices will provide extrusions between 400 and 1200 microns. The rolling process may be done as a batch step or as a continuous process in which extrusions are constantly fed from the extruder into the rolling apparatus. The rolled granules are then preferably dried.

In addition to the glyphosate and basic material, the water-dispersible granules of the invention will normally include a surfactant and optionally a carrier or filler. Examples of fillers include sand; clays and mineral earths such as attapulgite, kaolin, bentonite, fillers earth, keiselguhr, dolomite, talc, diatomaceous earth; fertilizers such as ammonium sulphate, ammonium phosphate and other organic and inorganic materials. The residual moisture of the dried granules will be in the range of about 0% to about 5.0%.

The following specific examples of water-dispersible granules are not meant to limit the invention.

EXAMPLE 7

	% w/w
Glyphosate (95% technical)	94.7
MORWET D425	5.0
Residual Water	0.3
Total	100%

MORWET D425 is a naphthalene formaldehyde condensate sodium salt.

EXAMPLE 8

	% w/w
Glyphosate (95% technical)	84.2
MORWET D-425	10.0
Talc	5.3
Residual water	0.5
Total	100%

EXAMPLE 9

	% w/w
Diammonium phosphate (98%)	81.65
GM 40 Clay (Kaolin clay)	10.00
Talc	8.00
Residual H_2O	0.35
Total	100%

EXAMPLE 10

	% w/w
Diammonium phosphate (98%)	81.65
Ultrazine NA	5.00
GM 40 Clay (Kaolin clay)	5.00
Talc	8.00
Residual H_2O	0.35
Total	100%

A preferred embodiment of the formulation comprises first a water-dispersible granule wherein the active ingredient is glyphosate and a second water-dispersible granule wherein the main ingredient is diammonium phosphate. The formulation further comprises a twin-pack which can be used by the end-user at the site of application and can further be combined with additional adjuvants.

The phytoactive compositions of this invention are effective when subsequently dissolved or dispersed in a suitable diluent, preferably water and applied to the locus desired by spray or other conventional means. Conventional adjuvants including wetting agents, penetrating agents, sticking or spreading agents and can be added to the final solution or dispersion.

The following examples demonstrate the herbicidal effectiveness of the invention.

EXAMPLE 11

This example demonstrates the post-emergence phytotoxic activity of the invention. The weed species used in this example were *Cynodon dactylon*, common name: bermuda grass and abbreviated CD; *Agropyron repens*, common name: quackgrass and abbreviated AGR; *Convulvulus arvensis*, common name: field bindweed and abbreviated COA; and *Cyperus rotundus*, common name: purple nutsedge, and abbreviated CYR.

Individual 6-inch diameter plastic pots were filled with sandy loam soil containing a commercial fungicide CAPTAN® and 17-17-17 fertilizer: ($N-P_2O_5-K_2O$ on a weight basis). Tubers of bermuda grass, purple nutsedge and quackgrass rhizomes were planted in individual containers. Sufficient stock was planted to produce several weed plants per container depending on the size of the plant. Ample seeds of field bindweed were also planted in individual pots. Six weeks after planting, the emerged seedlings of all species were sprayed with aqueous solutions of the test formulations. Additional untreated pots were used as standards for measuring the extent of weed control in the treated pots. Diammonium phosphate (DAP) was dissolved in 40 milliliters of the adjuvant solution and then added to glyphosate; after dissolving, the remainder of the adjuvant solution was added. All adjuvants were applied at 0.5% v/v.

The solutions were prepared to such dilutions that a spray rate of 25 gallons per acre gave from 0.125 to 1.000 pounds per acre of glyphosate as described for each test. Three replicate treatments were made for each glyphosate rate. The resulting solution was used for spraying at the rates indicated in Table 1.

Fourteen, 28 and 49 days after treatment, test pots were compared to the untreated pots and the weeds in each pot were rated visually in terms of percent desiccation ranging from 0% to 100%, with 0% representing the same degree of desiccation as the untreated check and 100% representing complete kill of all weeds. All types of plant injury were taken into consideration. The results are shown in Table 1 and 2.

TABLE 1

HERBICIDAL TEST RESULTS

1. TEST FORMULATION	APPLICATION RATE lb. AI/A	2. % DESICCATION DAYS AFTER TREATMENT 14 DAYS				28 DAYS				49 DAYS			
		CD	AGR	COA	CYR	CD	AGR	COA	CYR	CD	AGR	COA	CYR
Glyphosate	0.125	37	33	--	--	47	80	--	--	30	78	--	--
DAP	0.250	57	94	10	20	83	99	13	23	92	100	30	30
AL-2042	0.500	87	99	23	43	98	100	27	50	100	100	57	90
	1.000	--	--	50	73	--	--	60	90	--	--	90	100
Glyphosate	0.125	27	30	--	--	17	67	--	--	00	63	--	--
DAP	0.250	47	60	10	13	53	99	8	13	27	100	27	20
IGEPAL-DM-970	0.500	60	97	17	30	70	100	37	23	47	100	60	77
	1.000	--	--	33	53	--	--	80	80	--	--	73	100
Glyphosate	0.125	33	67	--	--	20	80	--	--	7	87	--	--
DAP	0.250	50	92	8	13	60	100	17	20	60	100	17	23
AL-2739	0.500	80	99	17	30	96	100	20	47	93	100	27	88
	1.000	--	--	27	63	--	--	37	97	--	--	67	100
Glyphosate	0.125	17	17	--	--	10	27	--	--	0	3	--	--
DAP	0.250	37	27	7	13	33	37	7	7	3	40	9	17
NB-01	0.500	57	94	23	33	67	100	10	27	27	100	30	80
	1.000	--	--	23	57	--	--	20	80	--	--	67	97

1. Glyphosate and DAP (diammonium phosphate) are applied at a 1:1 ratio.
2. The application rate is lb. glyphosate/acre.
3. -- indicates not rated.
4. AL-2042, AL-2739, NB-01, and IGEPAL-DM-970 are blended nonionic surfactants.

TABLE 2

HERBICIDAL TEST RESULTS

1. TEST FORMULATION	APPLICATION RATE lb. AI/A	2. % DESICCATION DAYS AFTER TREATMENT 14 DAYS				28 DAYS				49 DAYS			
		CD	AGR	COA	CYR	CD	AGR	COA	CYR	CD	AGR	COA	CYR
Glyphosate	0.125	27	67	--	--	87	90	--	--	70	80	--	--
DAP	0.250	53	99	13	20	97	100	24	37	99	100	31	3
AL-2042	0.500	73	99	33	47	99	100	53	77	100	100	88	27
	1.000	--	--	60	80	--	--	87	99	--	--	100	100

1. Glyphosate and DAP (diammonium phosphate) are applied at a 1:1 ratio.
2. The application rate is lb. glyphosate/acre.
3. -- indicates not rated.

EXAMPLE 12

The phytoactive effect of the extruded water-dispersible granules of glyphosate, Example 7 and diammonium phosphate, Example 9 was determined from broadcast application to established, non-crop field plots of quackgrass. The granules were applied with a CO_2 sprayer with 8002 Teejet nozzles at a volume of 250 L/ha. The plot size was 2 x 6 meters of silty loam soil type. Each treatment was replicate four times using a randomized block design. Application was at the 2-3 leaf growth stage at rates of 0.90, 0.71 and 0.53 kg glyphosate per ha at a 1:1 ratio with diammonium phosphate. The adjuvant, FRIGATE, a tallow fatty acid amine ethoxylate, was added to some of the formulations at 0.5% v/v upon application. At 5, 11 and 40 days after treatment, the weeds in each treatment plot were compared to the weeds in untreated plots and visually rated in terms of percent desiccation ranging from 0 to 100, with 0 representing the same degree of desiccation as the untreated check and 100% representing complete kill. The results are shown in Table 3.

EXAMPLE 13

The formulation of Example 11 was used to determine the phytoactive effect on Durum wheat var. Kyle. The granules were mixed with FRIGATE at 0.5% v/v and then sprayed with a backpack sprayer at a volume of 115 L/ha, with a pressure of 275 kPA.

Wheat was seeded at a rate of 90 kg/ha in field plots measuring 3 x 6 meters in sandy loam soil with 2% organic matter.

Each treatment was replicated three times in a randomized block design 46 days after seeding. The percent desiccation was evaluated by visual comparison to untreated plants 5, 14 and 28 days after treatment. The results are shown in Table 4.

TABLE 3

TEST FORMULATION	APPLICATION RATE kg/ha	% DESICCATION DAYS AFTER TREATMENT 5	11	40
Glyphosate	0.90	26	41	2
DAP	0.71	30	45	12
	0.53	23	30	13
Glyphosate	0.90	89	86	73
DAP	0.71	80	75	59
FRIGATE	0.53	82	82	67

TABLE 4

TEST FORMULATION	APPLICATION RATE kg/ha	% DESICCATION DAYS AFTER TREATMENT 5	11	40
Glyphosate	0.90	99	100	99
DAP	0.71	99	99	98
FRIGATE	0.53	92	93	96

The amount of formulation which constitutes a phytoactive amount depends on the nature of the plants and the effect desired. The rate of application generally varies from about 0.01 to about 50 pounds of glyphosate per acre, preferably from about 0.1 to about 25 per acre with the actual amount depending on the overall cost and desired results. It will be readily apparent to one skilled in the art that formulations exhibiting lower phytoactivity will require a higher application rate. In general, any conventional post-emergence method of application can be used.

Accordingly, the invention also includes a method of killing or controlling undesirable plants by applying an effective amount of the formulation of this invention to the locus of the plant or weed to be killed or controlled.

The specific embodiments described above should not be construed as a limitation on the scope of this invention.

WHAT IS CLAIMED IS:

1. A dry phytoactive water-soluble or water-dispersible formulation comprising the acid, N-phosphonomethylglycine and a basic material wherein the basic material is diammonium phosphate or guanidine salts.

2. A formulation of claim 1 which further comprises one or more surfactants.

3. A formulation of claim 1 or 2 wherein the formulation is a water-soluble or water-dispersible granule.

4. A formulation of claim 1 wherein the basic material is diammonium phosphate.

5. A formulation of claim 1 wherein the guanidine salt is guanidinium acetate.

6. A formulation of claim 1 wherein the formulation comprises a first granule of N-phosphonomethylglycine and a second granule of diammonium phosphate.

7. A formulation of claim 6 which further comprises one or more surfactants.

8. A formulation of claim 1 wherein the ratio by weight of N-phosphonomethylglycine to basic material is between about 10:1 to about 1:10.

9. A formulation of claim 8 wherein the ratio is between about 4:1 to about 1:4.

10. A formulation of claim 1 wherein the formulation contains water in the range from about 0.01 to about 8.0 percent by weight of the total formulation.

11. A formulation of claim 10 wherein the water is in the range of from about 0.01 to about 3.0 percent by weight of the total formulation.

12. A formulation of claim 2 wherein said surfactant comprises a nonionic surfactant, an anionic surfactant, a cationic surfactant, an amphoteric surfactant or mixtures thereof.

13. A formulation of claim 6 wherein the water-dispersible granules are prepared by mixing the N-phosphono-methylglycine into an extrudable form in the presence of water to form an extrudable wet mix, extruding the wet mix, rolling the wet extrusions to break down said extrusions to form granules and optionally drying the extrusions.

14. A formulation of claim 13 further comprising one or more surfactants.

15. A formulation of claim 14 wherein the surfactant comprises between about 0.1 to about 20% by weight of the total formulation.

16. A formulation of claim 13 wherein about 95% of the formulation comprises granules which will pass through a 1700 micron sieve but are returned on a 300 micron sieve.

17. A method of killing or controlling unwanted vegetation by applying to a plant or locus of the plant to be killed or controlled a phytoactive amount of the formulation of claim 1, 2, 6 and 13.

INTERNATIONAL SEARCH REPORT

Intern al Application No
PCT/GB 93/02204

A. CLASSIFICATION OF SUBJECT MATTER
IPC 5 A01N57/20 //(A01N57/20,59:26,47:44,25:32,25:14)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 5 A01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO,A,90 07275 (MONSANTO) 12 July 1990 see page 4, line 26 - page 5, line 4 see page 6, line 7 - line 19 see page 10, line 17 - page 11, line 4 see page 12, line 22 - line 25 see page 13, line 18 - line 25 ---	1-5, 8-12,17
X	WO,A,92 12637 (MONSANTO) 6 August 1992 see page 6, column 38 - column 8 see page 7, line 25 - line 34 see page 11, line 6 - page 12, line 5 see page 14, line 1 - page 17, line 2 see page 44, line 16 - line 27 --- -/--	1-5, 8-12,17

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
11 March 1994	25. 03. 94

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax (+31-70) 340-3016	Lamers, W

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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X	WO,A,91 08666 (CHEMINOVA AGRO) 27 June 1991 cited in the application see page 4, line 8 - line 13 see page 5, line 11 - line 27 see page 6, line 15 - line 24	1-4,8,9, 12,17
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X	EP,A,0 088 180 (GESHURI LABORATORIES) 14 September 1983 see page 3, line 19 - page 4, line 10 see page 5, line 1 - line 4 see page 13, line 15 - line 21 see page 18, line 4	1-3, 8-12,17
A	CHEMICAL ABSTRACTS, vol. 89, no. 19, 6 November 1978, Columbus, Ohio, US; abstract no. 158688c, D.J.TURNER ET AL. 'Complexing agents as herbicide additives.' page 169 ;column 2 ; see abstract & WEED RES. vol. 18, no. 4 , 1978 pages 199 - 207	1-17
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A	DATABASE WPI Week 8736, Derwent Publications Ltd., London, GB; AN 87-253854 [36] & JP,A,62 175 408 (ISHIHARA SANGYO KAISHA) 1 August 1987 see abstract	1-17

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